



# DATA VALIDATION REPORT

Gold King Mine Release Incident

SAMPLE DELIVERY GROUP: 680-117013-8

Prepared by

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## I. INTRODUCTION

Task Order Title: Gold King Mine Release Incident  
Project No.: 20408.012.001.0274.00  
20408.012.001.0267.00  
Sample Delivery Group: 680-117013-8  
EPA Project Manager: Steve Way  
Weston Project Manager: Dave Robinson  
TDD No.: 0001/1508-04  
Matrix: Soil/Water  
QC Level: Stage 2A  
No. of Samples: 2  
No. of Reanalyses/Dilutions: 0  
Laboratory: TestAmerica – St. Louis

**Table 1. Sample Identification**

<i>Location ID</i>	<i>Lab Sample Name</i>	<i>Matrix Type</i>	<i>Collection Date</i>	<i>Method</i>
CC06_092115_1300	680-117013-1	Solid	9/21/15 1:00 PM	903.0, 904.0, A-01-R
CC06_092115_1300	680-117013-3	Water	9/21/15 1:00 PM	903.0, 904.0, A-01-R





## II. Sample Management

Anomalies regarding sample management are noted below. According to the Login Sample Receipt Checklists, one of the samples was received below the temperature limits at  $1.3^{\circ}\text{C}$ ; however, as the sample was not noted to be frozen or damaged, no qualifications were required. The remaining sample was received within the temperature limits of  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . The samples were received intact and on ice. The chain-of-custody (COC) were appropriately signed and dated by field and laboratory personnel. The presence or absence of custody seals on the cooler was not specifically noted.

The following issues were noted:

- ☐ The soil sample was listed as CC06\_09212015\_1300 on the COC. A note from M. Pryhoda instructed the lab to log-in the sample according to standard Gold King Mine naming convention leaving off the 20 in the year.
- ☐ The original data package and electronic data deliverable (EDD) did not list the counting uncertainty or the total propagated uncertainty. The revised data package listing the measurement uncertainty was received by MEC<sup>X</sup> on 11/6/2015.
- ☐ Uranium and hexavalent chromium analyses requested on the COCs were reported in related SDGs 680-117013-3, 680-117-013-5, and 680-117013-7.
- ☐ The COCs did not list CLP sample IDs, and none were provided. The laboratory logged the samples per the location IDs on the COCs.
- ☐ The presence or absence of sample tags was not noted in the case narrative, and sample tags were not listed on the COCs.



**Data Qualifier Reference Table**

Qualifier	Organics	Inorganics
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit. The associated value is the quantitation limit or the estimated detection limit for dioxins or PCB congeners.	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit. The associated value is the sample detection limit or the quantitation limit for perchlorate only.
UB	The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination.	The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J+	Not applicable	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential positive bias.
J-	Not applicable	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential negative bias.





Qualifier	Organics	Inorganics
UJ	The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
UJB	The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."	Not applicable.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	Not applicable.
R	The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.	The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.



**Qualification Code Reference Table**

Qualifier	Organics	Inorganics
H	Holding times were exceeded.	Holding times were exceeded.
S	Surrogate recovery was outside QC limits.	The sequence or number of standards used for the calibration was incorrect
C	Calibration %RSD or %D was noncompliant.	Correlation coefficient is <0.995 or calibration was noncompliant.
R	Calibration RRF was <0.05.	%R for calibration is not within control limits.
B	Presumed contamination as indicated by the preparation (method) blank results.	Presumed contamination as indicated by the preparation (method) or calibration blank results.
L	Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.	Laboratory Control Sample %R was not within control limits.
L1	LCS/LCSD RPD was outside control limits.	LCS/LCSD RPD was outside control limits.
Q	MS/MSD recovery was poor.	MS recovery was poor.
Q1	MS/MSD RPD was outside control limits.	MS/MSD RPD was outside control limits.
E	Not applicable.	Duplicates showed poor agreement.
I	Internal standard performance was unsatisfactory.	ICP ICS results were unsatisfactory.
A	Not applicable.	ICP Serial Dilution %D were not within control limits.
M	Tuning (BFB or DFTPP) was noncompliant.	ICPMS tune was not compliant.
T	Presumed contamination as indicated by the trip blank results.	Not applicable.
+	False positive – reported compound was not present.	Not applicable.
-	False negative – compound was present but not reported.	Not applicable.
F	Presumed contamination as indicated by the FB or ER results.	Presumed contamination as indicated by the FB or ER results.
F1	Field duplicate results were outside the control limit.	Field duplicate results were outside the control limit.
\$	Reported result or other information was incorrect.	Reported result or other information was incorrect.





Qualifier	Organics	Inorganics
?	TIC identity or reported retention time has been changed.	Not applicable.
D	The analysis with this flag should not be used because another more technically sound analysis is available.	The analysis with this flag should not be used because another more technically sound analysis is available.
P	Instrument performance for pesticides was poor.	Post Digestion Spike recovery was not within control limits.
*II, *III	Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found.	Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found.





### III. Method Analyses

#### A. VARIOUS EPA METHODS —Radionuclides

Reviewed By: P. Meeks

Date Reviewed: October 22 & November 6, 2015

The samples listed in Table 1 for these analyses were validated based on the guidelines outlined in the *Sampling and Analysis Plan/Quality Assurance Project Plan for Gold King Mine Release, Silverton, San Juan County, Colorado* (2015), *United States Environmental Protection Agency Contract Laboratory Program Statement of Work for Organic Superfund Methods, EPA Methods 903.1, 904.0, and A-01-R*, and the *National Functional Guidelines for Inorganic Superfund Data Review* (2008).

- ☐ Holding Times: The samples were analyzed within 180 days of collection.
- ☐ Blanks: Rather than using the *National Functional Guidelines* to assess the method blank results, it was the reviewer's professional opinion the method blanks should be assessed using a standard radiological method, "Evaluation of Radiochemistry Data Usability" (Paar 1997). Detects listed in the table below determined to differ from the method blank detects at the 5% confidence level were qualified as estimated with a potential high bias (J+). Detects determined not to differ from the method blank detects at the 1% level of confidence were qualified as nondetected (UB), at the level of contamination. There were no other analytes detected in the method blanks.

Analyte	Aqueous Method Blank (pCi/l)	Soil Method Blank (pCi/g)	Qualified Sample
Thorium-230	0.1607 ± 0.131	0.1176 ± 0.0640	Both samples (J+)
Thorium-228	N/A	0.09403 ± 0.0651	Soil sample (UB)
Uranium-233/234	N/A	0.04890 ± 0.0458	Soil sample (UB)

- ☐ Laboratory Control Samples (LCS): The recoveries were within the laboratory control limits.
- ☐ Laboratory Duplicates: No laboratory duplicate analyses were performed on a sample in this SDG.
- ☐ Matrix Spike/Matrix Spike Duplicate (MS/MSD): MS/MSD analyses were performed on both samples in this SDG for all analyses. The recoveries were within the laboratory control limits. Radionuclides do not report relative percent differences (RPDs) for duplicate analyses. Instead precision is assessed via the relative error ratio (RER). The RER is the ratio between the difference in measured activities to the sum of the potential errors. The RERs were within the laboratory control limit of  $\leq 1$ .





- Field QC Samples: Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site samples. Following are findings associated with field QC samples:
  - Field Blanks and Equipment Rinsates: This SDG had no identified field blank or equipment rinsate samples.
  - Field Duplicates: There were no field duplicate samples identified in this SDG.



# Validated Sample Result Forms: 680-117013-8

*Analysis Method* 903.0

**Sample Name** CC06\_092115\_1300

**Matrix Type:** Solid

**Lab Sample Name:** 680-117013-1

**Sample Date:** 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
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Radium-226	T	13982-63-3	0.0339	1	0.0678	pCi/g	U	U	
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**Sample Name** CC06\_092115\_1300

**Matrix Type:** Water

**Lab Sample Name:** 680-117013-3

**Sample Date:** 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
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Radium-226	T	13982-63-3	0.534	1	0.107	pCi/L			
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*Analysis Method* 904.0

**Sample Name** CC06\_092115\_1300

**Matrix Type:** Solid

**Lab Sample Name:** 680-117013-1

**Sample Date:** 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
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Radium-228	T	15262-20-1	0.184	1	0.451	pCi/g	U	U	
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**Sample Name** CC06\_092115\_1300

**Matrix Type:** Water

**Lab Sample Name:** 680-117013-3

**Sample Date:** 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
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Radium-228	T	15262-20-1	0.335	1	0.487	pCi/L	U	U	
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*Analysis Method* A-01-R

**Sample Name** CC06\_092115\_1300

**Matrix Type:** Solid

**Lab Sample Name:** 680-117013-1

**Sample Date:** 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
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Thorium-228	T	14274-82-9	0.078	1	0.07	pCi/g		UB	B
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Thorium-230	T	14269-63-7	0.423	1	0.0323	pCi/g		J+	B
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Thorium-232	T	7440-29-1	0.0931	1	0.0426	pCi/g			
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Uranium-233/234	T	13966-29-5	0.078	1	0.0674	pCi/g		UB	B
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Uranium-235/236	T	15117-96-1	0.0247	1	0.037	pCi/g	U	U	
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Uranium-238	T	7440-61-1	0.034	1	0.0563	pCi/g	U	U	
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# Analysis Method A-01-R

**Sample Name** CC06\_092115\_1300 **Matrix Type:** Water  
**Lab Sample Name:** 680-117013-3 **Sample Date:** 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Thorium-228	T	14274-82-9	0.647	1	0.157	pCi/L			
Thorium-230	T	14269-63-7	0.855	1	0.225	pCi/L		J+	B
Thorium-232	T	7440-29-1	0.333	1	0.218	pCi/L			
Uranium-233/234	T	13966-29-5	2.96	1	0.225	pCi/L			
Uranium-235/236	T	15117-96-1	0.138	1	0.128	pCi/L			
Uranium-238	T	7440-61-1	2.74	1	0.165	pCi/L			